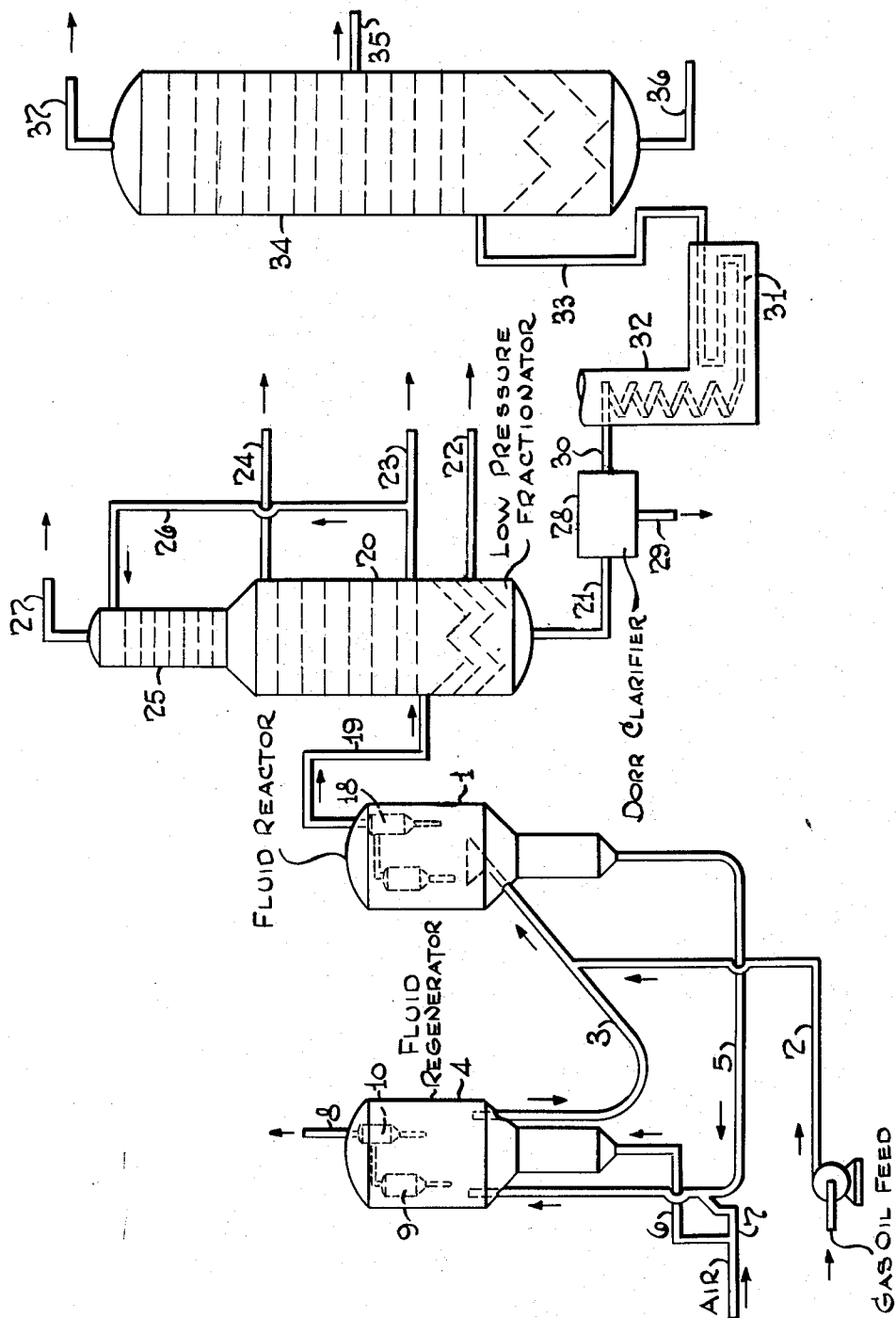


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BITUMINOUS SATURANT
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BITUMINOUS SATURANT

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This invention relates to an improved bituminous product for use in saturation of fibrous materials employed as structural forms for underground conduits, weatherboard, siding, insulating board. The saturant serves to improve the stiffness and durability of the fibrous material, and principally imparts resistance to water absorption and permeation which is necessary since the fiber board disintegrates in contact with water.

In recent years, suitable bituminous saturants meeting the requirements have been coal tar pitches and the tars available from the thermal cracking of gas oils. The coal tar pitches have never been available in adequate volume, and possess a strong odor which has severely limited their use for saturation of building materials such as weatherboard, siding and insulating board. With the advent of catalytic cracking processes, thermal cracking of gas oils has been essentially discontinued since the gas oils are the major feed stock for the catalytic process. This has resulted in a shortage of suitable saturant. Investigations have shown that the normal straight reduced or oxidized asphalts which can be prepared from bottoms obtained by distillation from crude oil, or the tars produced by light thermal cracking (visbreaking) of the crude bottoms are not generally satisfactory saturants since they either require relatively high temperatures or longer saturating times, or both, to effect satisfactory and ample saturation of the fibrous compositions.

It is accordingly the principal object of this invention to provide a process for preparing an asphaltic saturant which will yield an adequate supply of a saturant easily meeting trade specifications.

It is a further object of the invention to provide a process for the preparation of such a saturant which process overcomes the disadvantages pointed out above.

The foregoing and other objects are achieved, in accordance with the invention, by the thermal cracking of the cycle oil or clarifier oil obtained in a conventional fluid catalyst cracking operation and then distilling the tar fraction obtained to the desired consistency. By cycle oil or clarified oil is meant the heaviest material taken overhead from a fluid cracking reactor. Entrained catalyst accumulates in the heavy fraction and is separated therefrom by settling. The clarified oil thus obtained is then thermally cracked in accordance with this invention.

The type of bituminous material that can be employed as saturants is restricted to those materials which have suitable hardness at normal ambient temperatures in order to provide a saturated product with the proper rigidity, and which possess high susceptibility to tempera-

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ture so that the saturation can be accomplished rapidly and at lowest possible temperature. The permissible saturation temperature is particularly important since the major fibers used for these fabrications are organic in nature and cannot withstand pronounced exposure to high temperatures without charring, embrittlement or losing strength due to thermal degradation. Therefore, any successful saturating or impregnating process must be conducted at a temperature reasonably below the temperature of incipient disintegration and must be accomplished in as short a period as possible, not only to provide a more economical process, but to minimize the time at high temperature. It should be appreciated that time as well as temperature affect the degree of thermal disintegration. For example, with sugar cane fiber (bagasse), the incipient degradation temperature is about 475° F., but some degradation will occur if the fiber is held for one-half minute or longer at 450° F. Relative hardness is indicated by the A. S. T. M. penetration value of the bitumen at 77° F. Likewise, in order to insure saturation at temperatures that are not excessive, the viscosity at some elevated temperature—say, 275° F. or 300° F.—is normally specified. These two properties, penetration at 77° F. and Furol viscosity at 275° F., can be related by means of a fluidity index which is:

Fluidity index=

$$(\text{vis. at } 275^{\circ} \text{ F.} - \text{penet. } 77^{\circ} \text{ F.}) \times \frac{\text{pen. } 77^{\circ}}{100}$$

Saturants having a low index value are better suited for the purpose than those of higher index.

Typical examples of various bitumens, their fluidity index and saturating qualities are as follows:

Column	1	2	3	4	5
Bitumen	Tar from thermal cracked cycle stock	Tar from thermal cracked gas oil	Asphalt ¹		
			A	B	C
Softening point ° F.	140	143			
Penetration 77° F.	7	6	128	141	138
Furol vis.:			57	28	18
250° F.	132	171			
275° F.	54	67			
300° F.	36	43	365	500	290
Temp. ° F. for 50 furol	283	293	70	210	265
Fluidity index	3.3	3.7	314	364	390
Saturating ability: ²			8.6	176	132
350° F.	G	G	G	P	P
400° F.	G	G	G	P	F
425° F.	G	G	G	P	G
Min. temp. for good sat. ° F.	350	350	350-400	475	425

¹ Straight reduced or partially oxidized asphalts from Colombian or Californian crudes.

² Ability to yield a fiber board (bagasse type) with uniform surface saturation free from "fat" spots.

G=good; F=fair; P=poor (many fat spots).

It will be noted that the tars of columns 1 and 2 permit satisfactory saturation of bagasse type fiber board, i. e., a smooth, uniform and "dry" surface free of "fat" spots results when the board is dipped into a bath at the temperature indicated, for a period not exceeding 30 seconds.

The asphalts of columns 4 and 5, which represent the normal asphalts of the trade, saturate satisfactorily but at a higher temperature (425° F.). However, it will be noted that the hardness at ambient temperature, as indicated by the penetration at 77° F. is 18 or higher, which is not considered sufficient hardness to meet the requirements. Generally, a penetration 77° F. not exceeding 11 is needed. Thus, the only suitable products are those of columns 1 and 2. As previously stated, the tar from thermal cracking of gas oil is no longer available in reasonable quantity to meet the demand. However, the tar from the thermal cracking of the cycle oil from catalytic cracking has better over-all qualities than that from gas oil cracking, because the catalytic product is more aromatic in character. The fluidity index demonstrates this, the catalytic tar product showing an index not exceeding 4, whereas the gas oil tar generally has an index in the range of 8-12. Inspection of the indices of the asphalts from other crude sources also show that these products are high in index and less desirable.

For a further understanding of the invention reference may be had to the accompanying drawing in which the single figure is an elevational view generally diagrammatic, showing a flow plan embodying the improvement of the present invention.

Referring specifically to the drawing, a hydrocarbon petroleum fraction boiling in the gas oil boiling range (380°-1000° F.) is introduced into fluidized solids reactor 1 by means of feed line 2. Reactor 1 is filled with catalyst maintained by conventional means in a pseudo-liquid state by the incoming feed vapors. The feed is introduced into the catalyst line 3 connecting regenerator 4 with reactor 1. Temperature and pressure conditions in reactor 1 are adjusted so as to secure the desired conversion of the feed oil. Spent catalyst is withdrawn from reactor 1 by means of line 5 and passed into a fluidized solids regenerator unit 4 wherein temperature and pressure conditions are adjusted to secure the desired re-vivification of the catalyst. Air or oxygen-containing gas is introduced into regenerator 4 by means of lines 6 and 7. Combustion gases are withdrawn overhead from regenerator 4 by means of line 8. These combustion gases as they flow upwardly in zone 4 contain finely entrained particles of catalyst which particles are removed by means of cyclone separator units 9 and 10 disposed in the upper area of zone 4. The fluidized solids cracked products are removed overhead from zone 1 by means of line 19 and introduced into a distillation zone 20.

Temperature and pressure conditions are adjusted in zone 20 to remove a heavy fraction by means of line 21; a heavy gas oil fraction by means of line 22 and a light gas oil fraction by means of line 23. A fraction boiling in the motor fuel boiling range is removed by means of line 24. Gases are passed upwardly through an absorption section 25 and countercurrently contact a down-flowing absorption oil which is preferably separated from the light gas oil by means of line 26 and introduced into the top of the absorption section 25. Gas substantially completely free of

normally liquid hydrocarbons are removed overhead from zone 25 by means of line 27 and handled as desired.

The heavy fraction is withdrawn through line 21 and passed to clarifier 28 where entrained catalyst is settled out and removed through line 29. The clarified oil is withdrawn through line 30 and introduced into cracking coil 31 housed in furnace 32. Cracked products are withdrawn from furnace 32 through line 33 and introduced into distillation column 34 where temperature and pressure conditions are adjusted to remove a motor fuel fraction as a side stream through line 35 and a heavy tar fraction of the desired viscosity by line 36. Light gases are removed overhead through line 37. This heavy tar fraction is the saturant of the present invention. If desired the conditions in tower 31 may be so adjusted that the distillation is carried out under vacuum thus increasing the overhead yield and increasing the viscosity of the tar bottoms.

The fluidized solids technique for processing feed fractions, as for example, petroleum hydrocarbons and for carrying out other chemical reactions is a conventional one.

The conditions obtaining in the thermal cracking zone 32 are also conventional and are in the range between 850° F. and 1200° F. preferably between 900° F. and 1000° F. Pressures may vary from 300 to 400 lbs. per square inch to as much as 1000 lbs. per square inch.

Example

A heavy tar fraction obtained from a fluid catalytic operation and having a gravity of 19.2° API, 72.5% of which boiled above 700° F. was thermally cracked at 600 p. s. i. g. and 950° F. coil outlet temperature to produce 36 volume percent of gasoline having an end point of 430° F. and 62% of tar bottoms having a gravity of about 2° API. On vacuum reduction this tar gave a 26% yield of a saturant having 140° F. softening point and with excellent properties.

The nature of the present invention having been thus fully set forth and specific examples of the same given, what is claimed as new and useful and desired to be secured by Letters Patent is:

1. Process for preparing bituminous material, suitable as a saturant for fiber board and other purposes, which comprises subjecting the cycle oil from a catalytic cracking operation to thermal cracking said cycle oil having a gravity of 19.2 API, 72½% of which boils above 700° F. and then distilling the tar fraction to the desired consistency.

2. Process for the preparation of bituminous material, suitable as a saturant for fiber board and other purposes, which comprises contacting a hydrocarbon feed stream in a reaction zone in the presence of fluidized catalytic particles under temperature and pressure conditions adapted to crack said hydrocarbons, fractionating said cracked products to obtain a plurality of fractions including a high boiling fraction having a gravity of 19.2 API, 72½% of which boils above 700° F. subjecting the high-boiling fraction in the absence of catalysts to temperature and pressure conditions adapted to crack the same, distilling the resulting thermally cracked products to obtain a bituminous product having the desired viscosity.

3. Process as defined in claim 2 wherein the pressure in the catalytic cracking zone is in the range from about 0-30 lbs. per square inch gage

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and wherein the temperature in said catalytic reaction zone is in the range from about 800 to 1000° F. and wherein the pressure in said thermal cracking zone is in the range from 300 to 1000 lbs. and wherein the temperature in said thermal cracking zone is in the range from about 850° F. to 1200° F.

4. Process as defined in claim 3 wherein the feed stream introduced into said reaction zone is in the gas oil boiling range.

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